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Evolution of Efficient Methods to Sample Lead Sources, Such as House Dust and Hand Dust, in the Homes of Children[1]

SHANE S. QUE HEE,[2] BELINDA PEACE, C. SCOTT CLARK, JAMES R. BOYLE, ROBERT L. BORNSCHEIN, AND PAUL B. HAMMOND

Department of Environmental Health, University of Cincinnati Medical Center, Cincinnati, Ohio 45267-0056

Efficient sampling methods to recover lead-containing house dust and hand dust have been evolved so that sufficient lead is collected for analysis, and to ensure that correlational analyses linking these two parameters to blood lead are not dependent on the efficiency of sampling. Precise collection of loose house dust from a 1-unit area (484 cm^2) with a Tygon or stainless steel sampling tube connected to a portable sampling pump (1.2 to 2.5 liters/min) required repetitive sampling (three times). The Tygon tube sampling technique for loose house dust <177 μm in diameter was around 72% efficient with respect to dust weight and lead collection. A representative house dust contained 81% of its total weight in this fraction. A single handwipe for applied loose hand dust was not acceptably efficient or precise, and at least three wipes were necessary to achieve recoveries of >80% of the lead applied. House dusts of different particle sizes <246 μm adhered equally well to hands. Analysis of lead-containing material usually required at least three digestions/decantations using hot plate or microwave techniques to allow at least 90% of the lead to be recovered. It was recommended that other investigators validate their handwiping, house dust sampling, and digestion techniques to facilitate comparison of results across studies. The final methodology for the Cincinnati longitudinal study was three sampling passes for surface dust using a stainless steel sampling tube; three microwave digestions/decantations for analysis of dust and paint; and three wipes with handwipes with one digestion/decantation for the analysis of six handwipes together. © 1985 Academic Press, Inc.

INTRODUCTION

The relationship between lead contained in soil and in house dust and measures of lead exposure in children has been the subject of many scientific investigations in recent years (Angle *et al.,* 1974; Barltrop *et al.,* 1974; Barltrop, 1975; Brunekreef *et al.,* 1981; Charney *et al.,* 1980; Diemel *et al.,* 1981; Fairey and Gray, 1970; Lepow *et al.,* 1975; Milar and Cooney, 1982; Milar and Mushak, 1979; Roberts *et al.,* 1974; Roels *et al.,* 1980; Sayre *et al.,* 1974; Shellshear, 1973; Ter Harr and Aronow, 1974; Vostal *et al.,* 1974; Yankel *et al.,* 1977). Most of these investigators have tried to correlate soil or dust lead with blood lead with little success. There have been several reports on the correlation of hand lead to blood lead to support the hand-in-mouth route of lead ingestion (Charney *et al.,* 1980; Duggan, 1980; Lepow *et al.,* 1975; Roels *et al.,* 1980; Vostal *et al.,* 1974). Indeed, Buchet *et al.*

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[1] This paper was presented at The Second International Conference on Prospective Lead Studies, Cincinnati, Ohio, April 9–11, 1984.
[2] To whom correspondence should be addressed.

(1980), also found that near smelter areas, hand levels appeared to be correlated to body burdens of cadmium and arsenic.

The exact contribution of the hand-in-mouth route for lead absorption is dependent on many factors for each child; e.g., behavioral patterns, housing conditions, proximity of ultimate lead sources such as lead paint surfaces and fallout from automobile exhaust, behavioral patterns of the child, and the bioavailability of the ingested metal species. However, the representativeness of the environmental samples and validation of the sampling techniques for house dust and hand dust must be demonstrated before correlational results can be regarded as trustworthy. Previous studies have not documented these factors quantitatively.

Whether or not hand and blood lead are related may depend upon (1) the efficiency and representativeness of the washing or handwiping method, (2) whether the hands are deliberately washed by the child or by the caretaker prior to sampling (which is often beyond the investigators' control), (3) which parts of the hand are placed most often in the mouth, (4) whether the lead is evenly distributed over the surface of the hand, and (5) wiping varying from investigator to investigator. Vostal *et al.* (1974) reported even distribution of lead on the hands of inner-city children. Information on any of these factors was usually not provided in published studies.

The selection of sampling locations for house dust usually involves a somewhat subjective but not arbitrary procedure. X-Ray fluorescence data for painted walls are commonly used as a guide. In addition, the activity area of the child, the presence of lead-based paint, and the intactness of such surfaces, age and activity of the child, caretaker–child interactions, distance from roads, house cleanliness, ventilation, hermiticity of the home, playmate activities, and family schedules are some factors which should contribute to the selection of sampling locations. Once the sampling site has been located, the surface has to be sampled efficiently. The question of whether all or only part of the lead-bearing material on each surface should be or has been collected has never been demonstrated.

We undertook to investigate some of these sampling problems as part of a large prospective epidemiological study being conducted in Cincinnati, Ohio, where a primary source of the lead exposure for many children in this area is thought to be lead-based paint (Hammond *et al.*, 1980). We ultimately wish to establish correlations between lead in house and in outside dust, hand dust, and in paint with blood. This report concerns the evolution of representative and efficient sampling techniques for floor house dust and hand dust, as well as quick, efficient analytical techniques for determining the amount of lead in these sources.

MATERIALS AND METHODS

To develop methods of known efficiencies, the following steps were carried out: selection of representative dust, soils, paints, and surfaces; optimization of the analytical chemical method for lead in all of these matrices; and optimization and characterization of the sampling techniques for loose ("bioavailable" or "child available") floor and ledge dust, and hand dust.

Selection of Representative Samples of Soil, Dust, Paint, and Surfaces for Sample Collection

Soils. To ascertain whether geologic or anthropogenic sources of lead were present in the Cincinnati area, representative specimens uncontaminated by anthropogenic activity and from a specific prehistoric age were chosen to determine the crustal background level for lead. The Cincinnati area has six major native, geological soils:

Illinoian lake bed clay, weathered Illinoian red silt glacial till, and unweathered Illinoian glacial till, all from the Pleistocene era some 300,000 years ago. More ancient soils came from the Upper Ordovician era some 450 million years ago and include: fractured Kope formation shale soil, Fairview formation limestone soil, and Kope formation shale.

Approximately 1-kg samples from each location were gathered by a professional geologist, Dr. Warren Huff, University of Cincinnati, Department of Geology, who selected appropriate sampling locations for the collection of virgin samples free of anthropogenic contamination. The samples were dried in a dustless oven at 100°C until a constant weight was achieved. Each sample was then processed with mortar and pestle (metal-free porcelain apparatus) until all of the sample passed through a 149-μm brass sieve to produce reference house dust. Whitfield in 1979 defined this size fraction as a dust normally found in "clean room" environments. The sieved soil was tumble shaken for 1 day to ensure uniform mixing.

Dust. Preliminary observations suggested that the appearance of dirty hands was associated with dry, loose dust areas rather than with greasy floor areas or areas of dry, encrusted dust or mud. This loose dust would be mobile and could be associated with detection of high lead levels in dustfall containers.

Several houses were sampled for house dust obtained as vacuum cleaner bag collections. Objects such as twigs, glass fragments, insects, and paper were removed on passing through a 1-mm brass sieve. The dust was subfractionated through calibrated brass sieves to determine the particle size distribution. The fraction (approximately 1-kg) that passed through a 149-μm sieve was retained as loose reference house dust.

Paint. Four representative high-lead paints of different colors were obtained by mechanically stripping the paint, slicing it, processing with mortar and pestle (metal-free porcelain apparatus), and finally sieving all of it through the 149-μm brass sieve as above.

Representative surfaces were chosen to simulate those expected in study homes. The surfaces chosen were
   black painted wood table chipped and scarred (80 × 48 cm);
   top of cabinet black painted wood (36 × 63 cm);
   shelf fastened to a wall, white painted wood (60 × 25 cm);
   unpainted pine wood (17 × 18 cm);
   finished pine wood (31 × 15 cm);
   wood board with cracked green paint (29 × 35 cm);
   varnished wood board (28 × 28 cm);
   blue and white linoleum (30 × 30 cm);

dotted gray and white linoleum (30 × 30 cm);
rough surfaced white painted wooden board (12 × 12 × (cm);
indoor–outdoor carpet, mottled orange-yellow-brown-white (30 × 30 cm); orange plush pile carpet (30 × 20 cm).

Optimization of the Analytical Method for Lead in Paints, Soils, and Dusts

Before sampling methodology was evaluated, it was necessary to develop accurate, precise analytical methods for lead itself since lead may not be distributed uniformly through all substrate particle sizes.

The digestion method chosen was desired to be versatile enough to analyze the lead content of paints, soils, dusts, and handwipes. Among digestion mixtures evaluated were 1:1:1 $HF:HNO_3:HClO_4$(15 ml); 1:1 $HNO_3:HClO_4$ (15 ml); 3:2 $HNO_3:HClO_4$ (15 ml); 1:3 $HNO_3:HClO_4$ (10, 20 ml); 1:3 $HNO_3:HCl$ (10, 20 ml); 6:2:5 $HCl:HNO_3/HClO_4$ (10, 20 ml); nitric acid (10, 20 ml); and hydrochloric acid (10, 20 ml). All Teflonware (for HF digestions) and glassware were metal free (soaking overnight in 10% nitric acid followed by copious rinsing with distilled water). Environmental samples of 50 and 100 mg were allowed to stand in the acid solutions overnight in a fume hood, and the containers were covered with metal-free watch glasses (Teflon for HF-containing solutions). Digestion, with watch glasses on at 90°C on a hot plate, was continued until no brown nitrogen tetroxide was evolved (usually 1 hr). The supernatant was transferred to a metal-free beaker using a metal-free Pasteur pipet. The acid was then evaporated. Ten percent nitric acid (w/v) (10 ml) was then added with the beaker walls being irrigated, and the samples were refluxed gently (watch glasses on beakers) for 5 min. The dilute acid was removed by metal-free Pasteur pipets into calibrated graduated cylinders. The digestion/washing process was then repeated four more times using 2 hr digestions to ascertain which digestion composition was optimal and to determine how many digestions were necessary for quantitative recovery. The washings were analyzed by atomic absorption spectroscopy at 283.3 nm using an acetylene (4 liters/min)/air (18 liters/min) flame and the external standard method.

The 1:1:1 $HF:HNO_3:HClO_4$ mixture was utilized to ensure that any silicate matrix was disrupted and to produce the reference results.

Mixtures of specific paints, soils, and dusts in known weights were also analyzed.

After the optimal digestion mixture, number of digestions, and volumes had been ascertained, the optimum number of digestions was combined, the acid evaporated just to dryness, and the residue then redissolved in 10% nitric acid (10 ml) as above for atomic absorption spectroscopy (AAS) analysis.

To decrease analysis time, the CEM Corporation Microwave Drying/Digestion System (MDS-81) was utilized to digest dust samples. Since the digestions were performed in a perchloric acid fume hood, HCl could not be used. Thus, 10 ml of 5:4 $HNO_3:HClO_4$ was utilized in a program consisting of holding at 25% power for 10 min followed by 75% power for 10 min and finally 15 min at 0% power. The supernatant solution was then transferred. This was repeated two more times and all the supernatant solutions were combined before evaporation just to dryness. The residue was reconstituted as above in 10% nitric acid for lead analysis.

This microwave techn[ ]e allows quick sample turnaround time compared with a hot plate technique. Control digestion mixtures were also similarly analyzed.

Sampling Techniques: House Dust

The pump utilized was a calibrated battery-operated Bendix Corporation, Environmental Science Division Model BDX 30/31 personal sampler. Tygon S-50-HL tubing, 6.4 mm o.d., was used to connect the pump to the polystyrene sampling cassette, a 37-mm-diameter three-piece Aerosol Analysis Monitors cassette, preassembled with a thin cellulose support pad, 0.8-μm mixed cellulose ester plain white filter, and stoppers (Millipore Corp.), the three sections being held together with an outside cellulose band (Mine Safety Appliances, Part No. 625415) or parafilm wrap. Five-centimeter tubing (Teflon, Tygon, polyvinylchloride (3, 6, 9 mm i.d.)) was attached to the sampling port of the cassette to ascertain the best tubing diameter and material. The more inflexible tubing, e.g., Teflon and polypropylene, required a butt-to-butt joint made of Tygon to allow connection to the cassette. The sampling end of the tubing was cut at angles of 0, 30, 45, and 60° to ascertain the optimum surface contact angle for sampling.

The initial sampling efficiency determinations were performed on a smooth plastic container of dimensions 33 × 28 × 18 cm. Dusts (approximately 10, 20, 30, 40, 50, and 100 mg) were deposited as evenly as possible on the surface by passing the reference house dust through a 149-μm sieve while slowly moving the sieve 5 cm above the surface. The surface was then sampled at 2 liters/min by drawing the sampling tubing over the surface (with the thumb on the spine of the tubing) always in one direction, usually across the widest portion from left to right (if right-handed). This procedure was repeated five times. Times and weights of dust in the cassette and in the sampling tubing after each pass were noted as well as relative humidity, temperature, and atmospheric pressure. Thus, mass balance considerations allowed estimation of the sampling efficiency, the effect of surface loading, the optimum number of sweeps, and the effect of each pump. The amount of lead collected was used as a check.

Storage of sample. The collected dust samples were stored (stoppers on) with and without desiccating Drierite in quart-size Ziploc bags (Dow Chemical Co., Midland, Mich.) to assess storage stability. Cassettes without dust in them were also included as controls. The effects of storage intervals of up to 2 months were assessed by direct weighing.

Flow-rate dependence of sampling. Flow rates of 1, 1.25, 1.50, 1.75, and 2.0 liters/min were used on the surfaces to ascertain the influence of flow rate on the sampling efficiency.

Influence of surface type on sampling efficiency. The representative surfaces were then evaluated by the optimized sampling arrangement. As above, five separate sampling passes were made over each surface. After the fifth sampling pass, a final sampling pass with a 20-liter/min pump was performed. In some instances, five passes with the 20-liter/min pump were performed followed by one pass with the 2-liter/min pump. In another set of experiments, the sampling tubing was a 5 × 1-cm-i.d. piece of stainless steel 306, with the sampling end compressed to 1.5 cm width and 3-mm aperture. This was connected butt to butt to the cassette by

Tygon tubing. A weighed amount of reference house dust (around 50 mg) was applied to selected surfaces through a 149-μm sieve. The flow rate of the pump was 2.5 liters/min.

Influence of particle size of dust on sampling efficiency. Dust from a single household obtained by a vacuum sweeper was sieved into known weights of dust in the following diameter (μm) ranges: 44; 44–149; 149–177; 177–246; 246–392; and 392–833. Sampling was performed with the optimal technique validated on the plastic surface utilized in the first study given above after depositing 50 mg of dust into an area defined by a template of sampling area, 484 cm^2.

Influence of particle size on transfer of dust to the hand of small adults. Known weights (around 5 g) of sieved dry dusts in the particle size range given in the immediately preceding section were spread as evenly as possible in 7-mm-deep plastic petri dishes (20 cm^2). The hand of a small adult was then placed tightly over the dish, and the hand and the dish were inverted and then reinverted. The hand was removed from the dish so that nonadhering dust fell onto the weighing paper. The dust weight adhering to the hand was found by subtraction of the remaining weight.

Recovery of dust from sampling cassettes. To analyze the lead content in the collected dust, the dust was transferred from the cassette to the digestion beaker. The contents of the cassette were rinsed out by irrigation with wash bottles (three rinses) containing distilled water or 10% nitric acid. The water or nitric acid was then evaporated just to dryness at 100°C. The lead content was then found by digestion/AAS. The lead content of the same weight of dust contained in a beaker and analyzed by the same method was also found.

Representativeness of sampling house dust. A 1-unit square template (484 cm^2) was utilized to define the sampled area on floors in several homes where coverage of dust appeared typical and uniform to the eye. In one case, a 12-unit area was sampled in 1-unit increments on a carpeted surface, and the mean weights and lead of collected dust were compared.

Sampling Techniques: Handwiping

Handwiping for lead has been reported by Charney *et al.* (1980), Sayre *et al.* (1974), Vostal *et al.* (1974), and Chavalitnitikul *et al.* (1984). None of these investigations assessed or documented the efficiency of their methodology. Several wiping materials were evaluated in the present study: Wash n' Dry Soft Cloths Moist Towellettes No. 634910; Abco Nice Clean Towlettes; Washkin's Hospital Packettes; Walgreen's Brand Wet Wipes; Lehn and Fink's Wet Ones, and Baby Size Wet Ones. Hand rinses were also evaluated: 0.1 M HNO$_3$ (Roels *et al.*, 1980), and 0.01% (v/v) Zest soap solution. A combination of handwipes and rinsing was also evaluated. Three different types of house dusts were utilized.

For the validation studies in the laboratory, the human subjects (children 3 to 10 years old and adults) thoroughly washed their hands with soap and water, then blotted them dry on a paper towel. The hands were then rinsed with 500 ml 0.1 M HNO$_3$. The acid solution was poured slowly over all surfaces of both hands up to the wrists and the runoff solution collected by a large plastic funnel supported by a ring stand and positioned over the opening of a plastic sample bag

(quartz-size Ziploc) which was utilized to store the sample. The hands were again blotted. A weight (50 mg) of reference dust of known lead content was placed on the subject's palms with hands held over a plastic pan lined with waxed weighing paper. The dust was thoroughly rubbed onto all hand surfaces by rubbing the hands together for 30 sec. Nonadhering dust was caught by the waxed paper which was then weighed. The weight of adhered dust to the hand was then found by subtraction.

For handwipes, one towellette per hand was utilized. The towellette was unfolded. All hand surfaces up to the wrists and between the fingers were thoroughly wiped using a rotative motion. The towellette was then placed in the Ziploc quart-size bag for storage. The wipes were repeated five times. After the fifth wipe, the hand was rinsed with 0.1 M nitric acid as detailed above. For all these operations, the wiper wore gloves.

For hand rinses, the same apparatus was utilized as described above for the 0.1 M nitric acid wash. The 500-ml solution was slowly poured over all surfaces of both hands up to the wrists, or the solution was placed in the bag and the hand placed in the bag with the hand waved back and forth five times in the solution. This rinsing procedure was repeated four more times. After the fifth rinse, the hand was subjected to one wiping as detailed above for handwipes.

The rinses were transferred to a 250-ml beaker. The plastic bag was rinsed three times with deionized water, the washes were added to the beaker, and the aqueous solution was evaporated just to dryness. Unused bags were also similarly analyzed. The sides and bottom of the beaker were rinsed with 3 ml of 10% Ultrex nitric acid from a Pasteur pipet. The beaker (watch glass on) was then placed on a hot plate at 100°C for 2 min. The cooled solution was transferred to a 10-ml cylinder, the beaker was rinsed with 10% nitric acid, and the rinses were added to the cylinder. The volume was made up to 10 ml. The flask was shaken vigorously and analyzed for lead.

Soap solution rinsings and washings were evaporated in a 250-ml beaker on a hot plate at 100°C. Ten milliliters of 6:2:5 HCl:HNO$_3$:HClO$_4$ mixture was added to the cooled beaker, and the sample was left overnight. The sample was then heated for 1 hr at 100°C (watch glass on) and then evaporated just to dryness. Nitric acid (10% (v/v); 10 ml) was added, and the solution was gently heated at 100°C for 2 min and then transferred to a graduated cylinder for lead analysis. The washing process with 10% nitric acid was repeated two more times. Soap and acid blanks were also run.

Each handwipe was placed in a 50-ml beaker, the optimum digestion mixture (15 ml), 6:2:5 HCl:HNO$_3$:HClO$_4$ was added, and digestion was carried out at 100°C until no brown gas was evolved. The acid was then decanted into a beaker after centrifugation at 700 rpm for 10 min, the acid evaporated just to dryness, and the residue was solubilized in 10 ml of 10% nitric acid as described in the section on digestion. This procedure was repeated five times. This tedious process often took up to a week to complete. To shorten analysis time, the CEM microwave oven was utilized to carry out the digestion.

In the final method, each hand was wiped three times, and the six handwipes were placed in a 800-ml beaker to which 5:4 HNO$_3$:HClO$_4$ acid (100 ml) was

added. The microwave oven was operated at 10% power for 5 min and at 50% power for an additional 15 min, after which the contents were swirled manually and then processed again for 15 min at 50% power. The beaker was transferred to a hot plate and the acid just evaporated to dryness at 250°C. After cooling, the residue was redissolved in 10 ml 10% nitric acid as above for lead analysis.

RESULTS AND DISCUSSION

Analytical Methods for Dust, Soil, and Paint

All digestion mixtures gave the same lead concentrations for the particular paint, soil, and dust. Table 1 shows some sample results for nonmicrowave methods. Even in the 10% paint-in-soil mixture, the observed percentage of lead paint was analyzed correctly with the relative standard error (RSE) of 17% for 6:2:5 HCl:HNO$_3$:HClO$_4$, 3.3% for 3:1 HCl:HNO$_3$, and 3.7% for HNO$_3$. All these methods showed that paint and soil required three digestions/decantations, and dust required only one. Thus, the final method entailed performing three digestions/decantations for all samples. The only difference for the more reactive acid digestions, i.e., 1:1:1 HF:HNO$_3$:HClO$_4$ and 6:2:5 HCl:HNO$_3$:HClO$_4$, was that only two digestions/decantations allowed >99% recovery of all lead. One digestion/decantation for these digestion mixtures allowed >96% recovery. The microwave technique was limited by the fact that HCl could not be used since it would attack the perchloric acid fume hood. Nevertheless, for this technique, a 5:4 HNO$_3$:HClO$_4$ mixture gave the best results, but three separate digestions were still necessary to achieve >99% recovery of lead (Table 1). Lead values for all four representative paints examined (not all tabulated) using these techniques were essentially identical.

The analysis of lead of the native geological strata in the Cincinnati area resulted in the following (the data from the 1:1:1 HF:HNO$_3$:HClO$_4$ digestion are provided first, and data from 6:2:5 HCl:HNO$_3$:HClO$_4$ digestion are provided in parentheses. All data are expressed as micrograms Pb per gram soil): Illinoian

Lake bed clay, 16.4 ( ); weathered Illinoian red silt glacial till, 22.9 (12.1); unweathered Illinoian glacial till, 16.6 (10.5); fractured Kope formation shale soil, 22.2 (22.6); Fairview formation limestone soil, 19.2 (18.6); Kope formation shale, 14.2 (13.2). The relative standard deviations (RSD) of all the values were within 10%. The only two soils which gave differing results for the two digestion techniques were the unweathered glacial till and the weathered Illinoian red silt glacial till, for which the digestion mixture not containing HF was 63 and 53% efficient compared with a mean of 96.3 ± 2.1% for the other soils. Thus, the tills have more silicate-bound lead. The results also show little lead contamination occurred during the drying, sieving, and digestion processes. Hence, the native geological strata around Cincinnati do not exceed 23 µg Pb/g soil and contribute at least 11 µg Pb/g soil for all soil types. Therefore, lead values greater than 23 µg Pb/g in house dust must be related to anthropogenic activity rather than to dust from pristine dirts of the region. In both techniques, only two digestions and decantations were necessary to account for >99% of all lead. One digestion/decantation accounted for >99% of the lead for all samples except for fractured Kope formation shale soil (83%) and Fairview formation limestone soil (90%).

The reference house dust sieved into its constituent sizes (enumerated above in the section on *Influence of particle size of dust on sampling efficiency*) gave the lead content information contained in Table 2. The concentration of lead was generally independent of particle size, but most of the weight (ca. 75%) was contained in the fraction <149 µm. Thus, most of the lead was also contained in this fraction (77%). This supports the use of this size fraction as a reference house dust.

For handwipe materials, three successive digestions/decantations were required to recover >98% of the lead using the conventional hot plate wet digestions and the 6:2:5 HCl:HNO$_3$:HClO$_4$ mixture. The first digestion/decantation of a given handwipe material removed between 55 to 66% and two successive digestions/decantations, 89 to 91%. Thus, the multiple digestion technique allowed twice

TABLE 1

Comparison of Lead Recovery for Different Digestion Reagents for 50 mg of Three Paints, One Soil, One Housedust, and One Soil/Paint Mixture.[a]

Sample	Milligrams lead/gram substrate of lead[b] found from digestion with			
	6:2:5 HCl:HNO$_3$:HClO$_4$	3:1 HCl/HNO$_3$	HNO$_3$	5:4 HNO$_3$/HClO$_4$[d]
Paint 1	9.68 ± 1.20	10.4 ± 0.70	9.32 ± 0.80	—
Paint 2	1.29 ± 0.17	1.37 ± 0.07	1.23 ± 0.14	—
Paint 3	1.41 ± 0.20	—	—	1.40 ± 0.09
Soil 1	0.030 ± 0.008	0.032 ± 0.008	0.028 ± 0.008	—
10% mixture	0.868 ± 0.144	0.978 ± 0.032	1.08 ± 0.04	—
paint 1 in soil	(8.7 ± 1.4)%[b]	(9.4 ± 0.3)%[b]	(11.5 ± 4)%[b]	
Reference house dust	1.14 ± 0.15	1.10 ± 0.02	—	1.15 ± 0.08

[a] Detection limit of lead was 12 µg Pb/kg substrate.
[b] Calculated % paint in mixture.
[c] Mean ± SD of three replicate determinations.
[d] Microwave method.
—, not analyzed.

TABLE 2

House Dust Particle Size, Lead Content, and Sampling Efficiency for the Optimized Sampling Protocol.

Size range (µm)	Weight % of fractionated dust	Lead content µg Pb/g dust fraction	% Lead in unfractionated dust	Sampling efficiency for fractionated dust (%)
<44	18	1440	21	62
44–149	58	1180	56	76
149–177	4.5	1330	4.9	71
177–246	2.7	1040	2.3	47
246–392	6.1	1110	5.6	5
392–833	11	1090	9.6	14
Unfractionated dust	100	1214 ± 13[a]	100	62

[a] Standard deviation.

the efficiency of a single digestion. The same results were observed for the microwave method which utilized 5:4 HNO_3:$HClO_4$ and six handwipes (three from each hand). However, the recovery of spiked ionic lead in the first transfer was 89%. The major advantage of the latter technique was the rapidity of the analysis (4 hr versus 1 week for the conventional method). These results show that a single transfer using the acid solvent may not recover all the lead in a digested sample, even though recovery of spiked amounts of ionic lead may be acceptable.

Sampling Techniques: For House Dust

It was found that an angle of 45° for 3-mm-i.d. Tygon tubing was the optimal sampling configuration for passing the sampling tube over a smooth surface. Tygon was probably successful because of its flexible nature compared with polypropylene and Teflon. Furthermore, comparatively inaccessible surfaces such as window sills, corners, and ledges could be sampled as well as floors and other large flat surfaces. A 30° angle did not allow enough contact air volume to be pulled by the pump when the tubing was passed over the surface. A 60° angle did not allow sufficient contact of the pulled air with the surface to be sampled, resulting in inefficient sampling. A 0° angle did not allow a suction to be developed. The sampler did not sample dusts at all below a flow rate of 1.25 liter/min, and it was most efficient and reproducible at 1.75–2.0 liter/min, the flow rate here showing around 2% precision. The amount of dust retained in the Tygon sampling tube before the cassette was always around 4 to 6 mg. Thus, >80% collection efficiency for the cassette would require an original amount of dust to be sampled of at least 30 mg. This was also shown directly by experiment. The maximum amount that could be sampled was around 5 g. At cassette dust weights lower than 40 mg, the dust in the sampling tubing was weighed. Storage experiments revealed that dust could be stored for many months if necessary awaiting metal analysis. The changes in weight observed were caused by the progressive drying out of the cellulose shrink band binding the cassette together.

It was found that the filter itself did not collect all of the dust sampled, and in fact the collected weight on the filter was not proportional to the amount of dust sampled, the proportion varying from 6 to 15% (w/w) over a total sampled mass range of 50 to 100 mg. Most of the dust deposited on the sides of the cassette due to electrostatic attractions. Filter breakthrough as measured by placing another cassette behind the first one was less than 0.1% of the total amount of dust. Thus, a quantitative recovery technique had to include recovery of all the dust in the cassette and in some instances for collected weights less than 30 mg, in the Tygon tubing just before the cassette. The two washing techniques evaluated for dust recovery from the cassette and Tygon tubing before the cassette both gave 100% recoveries with, however, the water washing providing a relative precision of 10% rather than 36% as observed for the nitric acid washing medium.

The results for sampling loose dust on representative surfaces are shown in Table 3. These efficiencies are in relative weight percentage of recovered dust and define the cumulative recovery by the five wipes as 100%. In all cases, at least three sweeps were necessary to recover reproducibly at least 84% of the recovered dust for the 2-liter/min pump and also for the 20-liter/min pump. The

TABLE 3

DUST SAMPLING RECOVERY OF THE VACUUM METHOD USING THE OPTIMIZED TYGON TUBING SAMPLER AFTER SAMPLING VARIOUS SURFACES (CA. 900 cm^2) FOR DUST

| Surface | Pump | Cumulative recovery for sampling number | | | | |
		1	2	3	4	5
1	1	61	75	86	96	100
1	2	63	79	94	100	100
2	1	66	96	100	100	100
2	2	89	96	100	100	100
3	1	68	79	90	95	100
3	2	78	87	93	96	100
4	1	66	79	91	96	100
4	2	83	89	98	99	100
5	1	77	85	90	95	100
5	2	82	93	95	97	100
6	1	64	79	96	98	100
6	2	80	90	96	99	100
7	1	42	66	84	94	100
7	2	54	75	87	94	100
8	1	47	100			
9	1	73	100			
Average ± SD[a]	1	63 ± 11	80 ± 9	91 ± 6	96 ± 2	100
	2	76 ± 12	87 ± 8	95 ± 4	98 ± 2	100

Note. Pump 1, 2 liters/min. Pump 2, 20 liters/min. Key to surfaces: 1, Wood board, cracked green paint, 1 cm thick; 2, wood board, varnished, 2 cm thick; 3, linoleum, smooth but textured; 4, linoleum smooth; 5, wood board, white paint, rough; 6, carpet, indoor/outdoor; 7, carpet, shag plush pile; 8, wood, table black painted, chipped, and scarred; 9, top of cabinet, painted wood.
[a] Without surfaces 8 and 9.

recovery for three sweeps was 91 ± 6% (mean ± SD) for the seven surfaces relative to five sweeps of the 2-liter/min pump. The corresponding recovery for the 20-liter/min pump was 95 ± 4%, not significantly different. On a recovery basis, all surfaces gave statistically equivalent values for both pumps except for surface No. 7 (shag rug). For the 2-liter/min pump, the recovery for one sampling pass on surface 7 was only 42%, a reasonable result. Thus, one sweep was not sufficient to sample surfaces efficiently. For the 2-liter/min pump, the recovery for one sweep varied between around 42% (for shag rug) to 77% (for wood board), although the efficiency for surfaces 1 to 6 in Table 3 was 67 ± 5% with a RSD of 7.5%. In this case, for surfaces 1 to 6 correlations linking dust levels to other parameters (viz., blood lead) would not be changed since the precision of recovery is high in spite of nonoptimal efficiency. However, if the sampling efficiency is extremely variable, then the observed correlation with blood lead might be distorted or even understated.

Even for the 20-liter/min pump, the recovery for one sweep varied between 54% (for shag rug) to 89% (for varnished wood board); the recovery for surfaces

I to 6 for one sweep was 79 ± 9% with a RSD of 11%. Furthermore, the quantitative nature of the recovery with five sweeps by the 2-liter/min pump on surfaces 1 to 6 was demonstrated by the fact that negligible amounts of dust were found in a clean cassette when the surfaces were swept again by the 20-liter/min pump. The 20-liter/min pump had many disadvantages: it was noisy, heavy, and required line voltage. Furthermore, since the study homes very rarely had plush-pile carpet, the light, portable, quiet sampling pump was utilized with three sweeps of surfaces being performed (sampling time 5–7 min) over a 484-cm² area. For surfaces 1 to 6, the mean recovery for a three-sweep technique relative to five sweeps was 96 ± 3% (mean ±SD). This was not any better than for the 2-liter/min pump 92 ± 5%. The use of multiple sweeps nullified the dependence of the collection efficiency and precision on the type of surface sampled, at least for these surfaces. Few house lead studies have reported validation methods like those in the present study which ensure that sampling efficiency and precision are not important, uncontrolled variables when sampling surface dust.

The sampling results in Table 3 are for loose dust <149 μm applied on various surfaces. Use of loose dust simulated the most available dust surfaces to a child. The next steps were to assess if <149 μm was a realistic reference house dust particle size, to find if the lead concentration varied with particle size, and to characterize the sampling efficiency of the optimized sampling technique for loose dust of different particle sizes. These results are presented in Table 2. On a weight basis, 76% of the pooled house dust composite comprised particle diameters <149 μm. The fraction 149 to 392 μm constituted only 13% by weight of the total dust. The diameter range 392 to 833 μm comprised 11%. As can be seen, the sampling efficiency did not decrease until diameters of 177 μm were exceeded, and even the 177- to 246-μm fraction was sampled with 47% efficiency. Above 246 μm, the sampling efficiency was ≤ 14%. However, when the lead content, the amount of each size fraction, and the sampling efficiency are taken into account, the five-sweep sampling protocol for a 50-mg dust of size <149 μm would collect around 72% of the lead; for particles <177 μm, the efficiency was also 72%. The calculated overall efficiency for collecting lead in the unfractionated dust would be around 62%. However, with either three or five sweeps, the RSD would be less than 10%.

The next step was to assess if the sampling of a unit (484 cm²) area delineated by a Plexiglas template gave representative results for a typical carpeted surface. The twelve unit areas yielded a mean ± standard deviation of 28 ± 4 mg dust. Thus, sampling a unit area appeared to be representative.

All the above experiments involved the optimum Tygon tubing configuration. Results for the stainless steel tubing modification are provided in Table 4. In spite of the variation of surface types, the recovery results are similar to those for the optimized Tygon tubing sampler. Again, after three sweeps, the RSD in cumulative recovery was 6.4% (compare 6.6% for three sampling passes for the seven surfaces in Table 3 for the Tygon sampler). This confirms that repetitive sampling will provide precise results. However, the absolute recovery for the stainless steel sampler was 57 ± 14% for five sweeps compared with 72% efficiency from Table 2 for the Tygon sampler for the diameter fraction ≤149 μm.

TABLE 4

RECOVERY OF DUST FROM VARIOUS SURFACES USING THE VACUUM METHOD (2.5 liters/min) AND THE STAINLESS STEEL TUBING SAMPLER

| Surface | Weight applied (mg) | Cumulative recovery (%) for sampling number | | | | | Cumulative absolute efficiency[a] (%) |
		1	2	3	4	5	
1	53.8	73	90	96	97	100	81
	52.9	72	88	94	97	100	72
2	51.5	93	97	97	97	100	59
	51.5	86	95	98	99	100	66
3	51.9	82	99	100	100	100	47
	51.0	90	97	99	100	100	65
4	54.5	69	83	90	95	100	64
	54.8	72	88	93	98	100	47
5	54.3	52	70	83	91	100	56
	52.4	44	70	82	94	100	49
6	50.9	72	92	98	99	100	29
	51.6	80	92	98	99	100	52
Average ± SD	53 ± 1	74 ± 14	88 ± 10	94 ± 6	97 ± 3	100	57 ± 14

Note. Key to surfaces: Smooth—1, bare wood (324 cm²); 2, refinished wood (458 cm²); 3, linoleum (484 cm²). Rough—4, short carpet (484 cm²); 5, long carpet (484 cm²); 6, painted wood (484 cm²).
[a] Five runs.

Recovery of Lead from Hands

Table 5 shows the data for the recovery of lead from the hands of children of age 3 to 10 years and from adults using handwiping, handrinsing, and a combination method.

It is clear that results based on recovered hand lead instead of absolute lead recovery can be misleading, since 100% recovery with respect to recovered lead often did not coincide with 100% efficiency with respect to the known mass applied. Handwipe treatments 1, 2, 4, and 5 and handrinse treatment 8 were in this category. Handwipe treatments 3 and 6, handrinse 7, and the combination method treatment 9 were more efficient. However, the handwipe material used in treatment 3 became unavailable. For treatment 6, four wipes for children and adults were necessary to achieve 93 ± 4% (arithmetic mean ± standard deviation) recovery of recovered lead corresponding to 99 ± 26% absolute efficiency in the cases where known amounts of lead were applied. Three wipes for treatment 6 for all children and adults was 85 ± 7% efficient with respect to recovered lead; the RSD was 8% compared with 4% for four wipes. Either three or four wipes is probably acceptable. Table 5 also shows that the laboratory experiments dealing with known hand lead actually simulated the recovery of hand lead from children in high lead homes, where the quantity of hand lead and the form of hand lead was unknown.

The treatment 6 handwipe contained water, SD alcohol 40, propylene glycol,

TABLE 5
Lead Recoveries for Handwiping and Handrinsing Techniques for Hand Lead

Subject	Recovery method	Method	\% Cumulative apparent recovery at treatment					Cumulative absolute efficiency (%)
			1	2	3	4	5	
Adult	Wipe[a]	1	48	65	78	87	100	59
Adult	Wipe[a]	2	47	60	80	93	100	68
Adult	Wipe[a]	3	60	80	88	96	100	89
Child	Wipe[a]	4	82	100	100	100	100	16
Child	Wipe[a]	5	63	92	90	100	100	37
Child	Wipe[a]	6	58	77	91	97	100	61
Adult	Wipe[a]	6	58	73	78	91	100	106
Adult	Wipe[a]	6	67	88	95	97	100	78
Adult	Wipe[a]	6	63	75	80	89	100	98
Adult	Wipe[a]	6'	1	64	79	89	100	132
Adult	Wipe[a]	6[d]	72	87	95	97	100	121
Child	Wipe[b]	1	44	57	76	89	100	
Child	Wipe[b]	6	36	63	80	91	100	
	Wipe[b]	6	52	72	83	90	100	
	Wipe[b]	6	57	76	86	96	100	
Adult	Rinse[a]	7	61	85	92	97	100	74
Child	Rinse[a]	8	100	100	100	100	100	10
Adult	Rinse/wipe[a]	9						84
Average ± SD		6	52 ± 22	75 ± 9	85 ± 7	93 ± 4	100	99 ± 26

Note. Method. (1) Abco Nice Clean Towelette—benzalkonium chloride towelette No. 634910; (2) Washkins Hospital packette; (3) Wash 'n' Dri Soft Cloths Moist Towelettes; (4) Wet Ones (Lehn and Fink Co.); (5) Baby Size Wet Ones (Lehn and Fink Co.); (6) Walgreen's Brand Wet Wipes; (7) 0.1 M HNO₃; (8) 0.01% aqueous Zest solution; (9) 0.1 M HNO_3 followed by a handwipe with treatment 6 then followed by another rinse.

[a] 50 mg dust applied.
[b] Hand lead unknown initially.
[c,d] Dust 2 and dust 3, respectively, to assess recoveries for different housedusts.

sorbic acid, sodium nonoxynol-9-phosphate, Oleth 20, PEG/75, lanolin, disodium phosphate, citric acid, and fragrance. The area of its nonwoven fabric-like surface was 322 cm².

Most of the researchers who have published handwipe techniques have utilized only one wipe or one wash and have usually not provided documentation on the efficiency of their methodology. Even if the treatment 6 handwipe had been used by previous investigators in the same manner as in the present study, the absolute efficiency of lead recovery for one wipe would be 50 ± 29%, a most undesirable result. This figure was computed from Table 5 using the absolute recovery for one wipe as calculated from those experiments for which absolute recoveries were known. If one wipe of the handwipes utilized in treatments 1 to 5 were utilized, the equivalent figure would be 30 ± 15%, statistically indistinguishable from the results for treatment 6 because of the very poor precision. Thus, unless three to five wipes are performed, very poor precision in hand lead levels will result independent of the type of handwipe, and probably not enough lead will be collected for analysis purposes in many cases. In addition, the poor precision for one wipe means that correlational analyses will also have corresponding poor precisions, unlike in the house dust sampling technique where a good precision for one sampling was obtained in spite of an inefficient method.

When one handrinse with 0.1 M HNO₃ was performed, the absolute recovery was 45%. Thus, three to five rinses are again necessary to approach an absolute efficiency of 70%. This probably signifies that part of the dust is engrained in the hand and requires physical rubbing for removal. In all likelihood, one handrinse will have more precision than one handwiping, although this test was not explicitly performed. It is clear from Table 5 that recovery of hand lead depends not only on the type of handwipe or rinse but also on the number of repetitive wipes, the latter factor being most important to ensure that comparable efficiencies are attained for different hands. Since different investigators wipe with different pressures, clearly all investigators should validate their particular technique rather than assume that recovery is quantitative or that the amount detected is automatically "bioavailable."

The combination method gives a satisfactory recovery of 84%. However, since use of dilute nitric acid was not eagerly welcomed by the investigators for human studies, handwiping alone was the technique finally chosen.

The recovery of spiked ionic lead in the handrinse solutions was always 90 to 96%. The lead content of handwipe materials varied widely, reaching a high value of 5 μg Pb/g handwipe for the handwipes of treatments 4 and 5. The handwipe for treatment 6 generally had a lead value of <2 μg Pb/g handwipe.

Dependence of Adherence of Dust on Particle Size

Once an adequate method (handwiping; treatment 6) had been found for recovery of hand lead, it was possible to assess if there was a particle size dependence of adherence of dust to the palms of the hands. These results are provided in Table 6. When the two dusts of largest diameter (>246 μm) are excluded, the

TABLE 6
Adherence of Applied Dust (around 5g) of Known Particle Size to the Palm of a Small Adult Hand

Diameter (μm)	Weight adhering to hand (mg)	% Adherence
<44	27.2	0.5
44–149	27.7	0.2
149–177	31.5	0.8
177–246	29.2	0.8
246–392	10.3	0.2
392–833	61.4	1.3
Arithmetic mean ± SD	31.2 ± 16.6	0.63 ± 0.42
	28.9 ± 1.9[a]	0.58 ± 0.29[a]

[a] Without the data above 246 μm.

average adherence is around 0.58 ± 0.29%, or the maxir   capacity of each hand is 28.9 ± 1.9 mg. Since most dusts <246 μm in our study houses contained 83% of the weight and 85% of the lead (Table 2), the potential hand lead for a dust which contains 1 mg Pb/g dust could be a maximum of 25 μg lead.

Values of up to 250 μg Pb recovered from both hands of our study children imply a minimum Pb content of around 4.3 mg Pb/g dust assuming equal lead distribution for both hands and that the hand is the same size as our adult subject; that is, the maximum capacity is 29 mg dust/hand. If one finger represents 12% of the area of a hand containing 500 μg Pb, the amount of lead recovered from the finger could be up to 20 μg assuming equal coverage. If it is known from observation which part of a hand the child favors licking, then the dust on this area can be sampled quantitatively using the multiple wipe technique or can be estimated assuming equal coverage if not enough lead can be collected.

Sampling of House Dust on Greasy Surfaces

The method for dust sampling discussed above allows precise sampling of loose dust <177 μm in diameter, which appears to comprise 81% by weight and 82% of the lead contained in typical dusts from houses of children (Table 2). To recover larger diameter and ingrained dust, we applied our handwiping technique (five wipes) to the floors just sampled by our optimized method for loose dust. One wipe was also not sufficient to sample this type of surface. Obviously, each surface will have its own type of lead loading, and sampling decisions for surfaces with greasy dust and encrusted mud will depend on the state of the deposit. Quantitative sampling of lead in greasy surfaces presents a research problem of considerable difficulty. In any event, lead in loose dust is probably more available to the child than in greasy deposits or encrusted mud. The vacuum method is still applicable.

The results of the efforts in our full study to correlate blood lead with hand lead and house dust lead are beyond the scope of this paper and will appear elsewhere.

CONCLUSIONS

If a sampling method has the same efficiency over all surfaces likely to be encountered in a study, then correlational analyses will not be affected unless the minimum detection level is not exceeded. One wipe recovered 1 to 82% of the hand lead with a RSD of 42 to 110% irrespective of the type of handwipe. In contrast, one sampling pass with the Tygon tube sampler for loose house dust with a 2-liter/min pump recovered 63 ± 11%, and one pass with a 20-liter/min pump recovered 76 ± 12% on the surfaces tested. Since the precision of sampling hard, smooth surfaces was generally acceptable, valid correlations would be obtained in spite of the relatively poor sampling efficiency. This would not be so, however, if not enough dust were collected to exceed the detection limit or if the sampling efficiency were markedly dependent on the type of surface. Since the detailed aspects of sampling protocols, details on types of surfaces, and a surface-specific compartmentalization of environmental data are rarely attempted or provided in published studies, the true correlations between blood lead, hand lead, and house dust lead can be distorted by the variation inherent in the sampling

technique. The biolog   meaning of the levels found on the floor and on the hand depend on the si ..on. Thus, the child may prefer one area to others and may suck a preferred finger or part of the hand. Use of the strategies presented here will maximize the likelihood of detecting the suspected toxicant once hygienic and preferred area considerations are taken into account.

The choice as to which house dust sampler to use, the Tygon or stainless steel, does not arise if correlations alone are being considered since both sample precisely if the repetitive sampling technique is employed. As to the significance of the absolute levels, the type of surface determines the significance of the data. Optically smooth surfaces are probably best sampled for available dust by the stainless steel sampler since its sampling geometry and the surface are relatively constant. If the surface has cracks or significant steep depressions into which fingers of children could be poked, or if the surfaces have corrugations, e.g., some window sills, then the Tygon tubing sampler is probably favored since Tygon is flexible enough to reach such places. The stainless steel sampler was the one chosen for the surfaces in our study homes.

This study has shown that much variation can be caused by not choosing a sampling method with a sampling efficiency independent of surfaces or by not being able to collect enough lead for analysis. The house dust samples were loose and relatively available to the child. It was demonstrated that loose dust particle sizes ≤246 μm could adhere to a child's hands. The study also showed that loose house dust and loose hand dust could be sampled adequately by at least three repetitive samplings. The techniques developed here are also potentially useful for sampling house dust in homes near smelters emitting arsenic, cadmium, and other nonvolatile toxic compounds in aerosol form. The strategies for establishing sampling efficiencies are also pertinent to other areas: forensic chemistry, e.g., for handwiping hands that contact a gun, or assay dust and soils for paint chips from car accidents; in occupational hygiene, for example, in foundries; and environmental chemistry, e.g., assessing probable exposure of children who mouth dioxin-contaminated soils. In any event, it is likely that a multiple wipe methodology will have to be used for effective and sensitive sampling.

The preceding results have demonstrated that house dust and hand dust sampling techniques have to be validated for each investigator before homogeneous data independent of sampling bias can be obtained. The literature contains little documentation of the efficiencies of handwiping or house dust sampling techniques. A very recent report documented that the Occupational Safety and Health Administration (OSHA) wipe test for lead-containing dust <40 μm diameter using filter paper varied in recovery between 31 to 212% for different wipers on the same smooth surface (Chavalitnitikul and Levin, 1984). Moistened filter paper or paper towels were more effective 89 ± 2 (mean ± SD) and 79 ± 5%, respectively. For porous surfaces like plywood, the filter papers and paper towels allowed only 38 ± 5 and 35 ± 5% recovery, respectively. Adhesive surfaces were more efficient for plywood (64 ± 13%). These investigators also utilized handwiping and facewiping to assess if blood lead could be correlated to hand and face lead. They did not validate these techniques (Chavalitnitikul et al., 1984).

Since investigators have their own techniques and can wipe more or less in-

tensely than in the present study, it is imperative that investigators should validate their individual techniques. For our particular methodology, three sampling sweeps for house dust, three handwiping passes, three digestions/decantations for dust, soils, and paint, and one digestion/decantation for handwipes using a microwave oven technique were adopted. These were the methodologies selected for the longitudinal study.

ACKNOWLEDGMENTS

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